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(54) Title: ACETALS AS INSECT REPELLANT AGENTS

(54) Bezeichnung: ACETALE ALS INSEKTENABWEHRMITTEL

(57) Abstract: The invention relates to a novel insect repellent agent, comprising an acetal as active component.

(57) Zusammenfassung: Die vorliegende Erfindung betrifft ein neuartiges Insektenabwehrmittel, das als aktive Komponente ein Acetal enthält.

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## Acetals as insect repellents

This invention relates to an insect repellent to repel flying, stinging, biting and sucking insects as well as pests of the genus Acarina (mites and ticks).

### Description of the state of the art

Insect repellents are chemical substances which have a repelling or destructive effect on insects and Acarina. Of great practical importance is their use in human and veterinary hygiene where they protect persons and animals from attack by blood-sucking, stinging, biting pests which are not just annoying, but are also potential carriers of diseases (malaria, spring-summer encephalitis, Lyme disease and many others). With insect repellents which are applied directly to the skin, it is necessary that they are compatible with the skin, non-toxic, proof against sweat and light and that there are no problems with regard to cosmetics (do not affect the skin, do not cause drying or wrinkles when applied externally) and pharmacological health aspects (irritation, penetration of deeper skin layers and in the blood and lymph circulation). In addition the protection of the skin sections to be treated or the protection of persons and animals due to treated objects in the surroundings should be retained as long as possible and the effective spectrum of the insect repellents should be as large as possible, i.e. they should be effective against as many different pests as possible.

In the past and to a lesser degree to the present time distilled oils, such as citronellol and lemon grass oil, as well as cloves oil, lavender oil, and eucalyptus oil and camphor have and are used as repellents, but which however all exhibit severe disadvantages, such as for example:

- questionable contents, such as eugenol in cloves oil, which in animal experiments has been found to be a carcinogen, mutagen and a skin-irritant or

cineole in oil from *Eucalyptus globulus*, which, when applied to the skin, is able to cause strongly irritating exanthema;

- only short-term effectiveness, because the distilled oils evaporate quickly due to the body temperature of the skin surface and consequently frequent follow-up treatment is necessary to ensure protection;
- completely lacking resistance to light and consequently there is the permanent risk of photosensitization and product modification already before application.

In the recent past these compounds have therefore largely been superseded by so-called synthetic repellents. In the state of the art applied synthetic repellents are, for example, phthalic acid dimethylester, 1,2-ethylhexane-1,3-diol, 3,4-dihydro-2,2-dimethyl-4-oxo-2H-pyran-6-carboxylic acid n-butylester, succinic acid dipropylester, N,N-diethyl-3-methyl-benzamide (DEET – also N,N-diethyl-m-toluamide) and pyridine-2,5-dicarboxylic acid di-n-propylester (Ullmanns Encyclopädie der techn. Chemie, 4th edition, Vol. 13, p. 237 ff., 1977). Recently, hydroxyethylbutylpiperidine carboxylate [1-piperidine carboxylic acid-2-(2-hydroxyethyl)-1-methylpropylester] has been used increasingly.

Often however, these synthetic repellents are not resistant to sweat, irritate the mucous membranes and are also able to penetrate the uppermost layer of the skin, accumulating in the body, whereby the side effects resulting from this have not yet been completely researched, but there is a justified suspicion of toxic effects.

### **Object of the invention**

Accordingly, the object of this invention is to provide an effective insect repellent for application directly onto the skin and / or the clothes and / or other objects and materials (bed linen, tent awnings, table cloths, etc.) surrounding the user requiring protection from annoying, harmful or disease-carrying stings and bites from insects and acarids (in particular ticks), based on natural raw substances or substances identical to natural

substances, with the lowest toxicological risks, which in addition develops a high effectiveness over as long a time period as possible.

### **Brief description of the invention**

This object is solved according to the invention by an insect repellent according to Claim 1 or by the use according to Claim 2. Preferred embodiments are given in the sub-claims. The basic structure of the substances according to the invention is an acyclic terpene with ten carbon atoms, i.e. a monoterpene, preferably singly or doubly unsaturated. The organic groups forming the acetal or semi-acetal are termed in the following as acetal or semi-acetal radicals.

The following description explains this invention in more detail by reference to the insect repellent according to the invention. These comments also apply in equivalent form to the claimed usage.

The effectiveness of the compounds according to the invention is based on their capacity of forming a fully covering, long-lasting film on the skin, which reliably prevents the substances or signals (sweat, CO<sub>2</sub>, uric and butyric acids, body heat) essential for the location of suitable objects of prey (people and mammals) being released from the skin for biting and stinging, blood-sucking pests, so that a warm-blooded object treated with the compounds according to the invention can no longer be identified and is therefore not subject to attack. With contact or direct action the agent according to the invention has a mortal effect due to the properties of the insect repellent according to the invention in that the chitin layer of the pests is modified and fluid is forced from their bodies. These direct actions are of an essential purely physical nature, so that the development of resistances, as inevitably occurs with chemically or biochemically effective substances due to the rapid alternation of generations and thus the rapid passing of the appropriate genetic information to the descendants is eliminated with the substances employed according to the invention. The use according to the invention of

the substances quoted in this application ensures a good insect repellent effect. The application can either take place directly on the skin of the individual to be protected (person or animal) or also to the clothing or other articles, such as bed linen, etc.

### **Brief description of the figures**

Figures 1 to 63 show the chemical composition of some of the compounds quoted in the description, which are particularly preferred in the practice of this invention.

### **Detailed description of the invention**

The substances according to the invention which are used as an insect repellent, such as for example the following preferred substances

Octadienal dialkylacetal,	Octenal dialkylacetal,
Octadienal octadienyl acetal,	Octenal octadienyl acetal,
Octenal octenyl acetal,	Octadienal octenyl acetal,
Octadienal-p-menthenyl acetal,	Octenal-p-menthenyl acetal,
2-heptadienyl-1,3-dioxane,	2-heptadienyl-1,3-dioxol,
2-heptenyl-1,3-dioxane and	2-heptenyl-1,3-dioxol

can be directly procured if they are commercially available or they can be produced from the following compounds, whereby the principal reaction steps are known to the person skilled in the art:

Constituents of natural distilled oils or their synthetically manufactured substitutes, such as:

- -neral and geranial [3,7-dimethyl-2,6-octadienal],
- (+)-citronellal, (-)-citronellal [3,7-dimethyl-6-octenal],
- geraniol [(E)-3,7-dimethyl-2,6-octadien-1-ol],
- linalool [3,7-dimethyl-6-octadien-3-ol],
- nerol [(Z)-3,7-dimethyl-2,6-octadien-1-ol],
- (+)-citronellol, (-)-citronellol [3,7-dimethyl-6-octen-1-ol] and
- (+)-terpineol, (-)-terpineol [p-menthane-1,8-diol]), which are reacted amongst themselves and also using mono- or multivalent, primary, secondary or tertiary, straight chained, branched, saturated or unsaturated alcohols with one to fifteen C atoms according to methods described below to the respective acetals.

Organic or inorganic acids are used as catalysts.

Preferred acetals are those whose acetal groups are formed symmetrically by alkyl groups with one to fifteen carbon atoms, more preferably with one to twelve carbon atoms. The quoted carbon atom numbers also apply to the semi-acetals.

The compositions according to the invention can contain other substances such as: saturated or unsaturated, aliphatic carboxylic acids with one to twelve carbon atoms, preferably octanoic acid or decanoic acid, benzoic acid ester, p-mentha-3,8-diols, hydroxyoctanals, p-mentha-3,8-diylacetals.

Preferred compositions according to the invention contain:

0.02 to 95.00% wt. of one or more of the substances according to the invention, preferred structures 1 to 44 (in the following (I)),

0.00 to 95.00% wt. of a natural or synthetic fatty oil in which the substances according to the invention are soluble, preferably coconut oil (in the following (II)),

0.02 to 50.00% wt. of one or more saturated or unsaturated, aliphatic carboxylic acids with 1 to 12 carboxylic atoms, preferably octanoic or decanoic acid (in the following (III)),

0.02 to 50.00% wt. of one or more benzoic acid esters preferably with the Structure 45 to 51 (in the following (IV)),

0.00 to 50.00% wt. of p-mentha-3,8-diol, preferably with the Structure 52 or 53 (in the following (V)),

0.00 to 50.00% wt. of hydroxyoctanal preferably with the Structure 54 to 55 (in the following (VI)),

0.00 to 20.00% wt. of one or more diylacetals, preferably with the Structure 56 to 59 (in the following (VII)),

0.00 to 50.00% wt. of one or more emulsifiers, preferably lecithin, PEG derivatives of castor oil and / or decylglucoside (in the following (VIII)),

0.00 to 99.88% wt. of water (in the following (IX)).

More preferably a composition according to the invention contains:

0.02 to 30.00% wt. of I

0.05 to 30.00% wt. of III

0.08 to 30.00% wt. of IV

5.00 to 30.00% wt. of V

0.05 to 10.00% wt. of VII

0.05 to 10.00% wt. of VIII

5.00 to 99.88% wt. of IX

Especially preferably a composition according to the invention contains:

0.02 to 16.00% wt. of I

0.05 to 20.00% wt. of III

0.08 to 10.00% wt. of IV

0.05 to 10.00% wt. of VIII

44.00 to 99.88% wt. of IX

The insect repellents according to the invention can be incorporated into the following types of finished products to give the consumer a choice in the form of the applied products:

**Emulsion, dispersion, lotion, cream, gel or solution.**

All the usual methods of preparation can be used for the manufacture of these forms of application.

Similarly, the usual basic materials and additives can be used. These comprise solvents, solvent accelerators, solubilisers, emulsifiers, wetting agents, anti-foaming agents, halogens, buffers, gel and film promoters, thickening, binding and lubricating agents, greasing agents, spreading agents, anti-sticking and flow regulation agents, moisture retention and drying agents, acid and alkali pH regulators, such as organic acids and fruit acids or hydroxides of earthy base metals, amines and amides, fillers and auxiliary substances, such as antioxidants, preservatives, odour adjusters and colorants, which are usually used for chemical-technical, cosmetic and pharmaceutical preparations.

The term "emulsion" comprises all dispersive systems of two or more liquids mutually immiscible, whereby the emulsion components may also be present at room temperature as solids or amorphous and crystalline waxes. These emulsions may be macro or micro-emulsions. Typically water-in-oil and / or oil-in-water emulsions are used. Emulsifiers are used to reduce the interfacial work (work to be applied in emulsifying). Emulsifiers are normally interfacially active substances, typically with hydrophilic end groups. Typical examples of these comprise:

- anionic emulsifiers, i.e. emulsifying agents with carboxylate, sulphonate, phosphate, polyphosphate, lactate, citrate, tartrate, glucose or polyglucose end groups;
- cationic emulsifiers, i.e. emulsifiers with amine salts or quaternary ammonia end groups;



- amphoteric and zwitter ionic emulsifiers, i.e. emulsifiers with zwitter ionic or betaine end groups, as well as
- non-ionic emulsifiers, i.e. emulsifiers with alcohol, polyether, glycerine, sorbit, pentaerythrite, saccharose, acetic acid and / or lactic acid radicals in the end group.

All emulsifiers contain in addition lipophilic end groups, such as alkyl or alkyl radicals, appropriately straight chained, branched or cyclic, as well as aryl and alkylaryl radicals. Furthermore hydrophilic side groups can be included such as hydroxyl, ester, sulphamide, amide, amine, polyamide, polyamine, ether, polyether, glycerine, sorbit, pentaerythrite or saccharose groups.

The term "gels" comprises dimensionally stable, easily deformable systems, rich in liquids, of at least two components. Normally these two components are: a) a liquid and b) a solid, colloiddally distributed substance, such as gelatine, silicic acid, montmorillonite, bentonite, polysaccharides, polyacrylates, pectins and many more.

The methods for the manufacture of all insect repellents according to the invention can be carried out in any production facility equipped for the production of chemicals without special technical effort.

The following examples are quoted as typical methods:

The constituents are stirred in an aqueous solution for 2 to 15 hours at 50°C to 150°C in a suitable, tightly sealed stainless steel vessel with a minimum quality of V4A with permanent stirring mechanism and heating.

The pure acetals are powders or crystalline, but fat-dispersing substances so that use in a pure form as insect repellent for the skin is difficult. Coconut oil has proven to be a good dispersion agent. The addition of the foodstuff emulsifier lecithin can be used for the production of aqueous solutions.

The following compositions were produced by suitable mixing of the quoted compounds:

IAM 1 = 95.00% wt. Structure 1 + 5.00% wt. coconut oil

IAM 2 = 5.00% wt. Structure 1 + 95.00% wt. coconut oil

IAM 3 = **50.00% wt. Structure 1 + 15.00% wt. coconut oil + 10.00% wt. lecithin +  
25.00% wt. water**

IAM 4 = 50.00% wt. Structure 2, ditto

IAM 5 = 50.00% wt. Structure 3, ditto

IAM 6 = 50.00% wt. Structure 4, ditto

IAM 6 = 50.00% wt. Structure 5a, ditto

IAM 7 = 50.00% wt. Structure 5b, ditto

IAM 8 = 50.00% wt. Structure 6a, ditto

IAM 9 = 50.00% wt. Structure 6b, ditto

IAM 10 = 50.00% wt. Structure 7a, ditto

IAM 11 = 50.00% wt. Structure 7b, ditto

IAM 12 = 50.00% wt. Structure 8a, ditto

IAM 13 = 50.00% wt. Structure 8b, ditto

IAM 14 = 50.00% wt. Structure 9a, ditto

IAM 15 = 50.00% wt. Structure 9b, ditto

IAM 16 = 50.00% wt. Structure 10a, ditto

IAM 17 = 50.00% wt. Structure 10b, ditto

IAM 18 = 50.00% wt. Structure 11a, ditto

IAM 19 = 50.00% wt. Structure 11b, ditto

IAM 20 = 50.00% wt. Structure 12a, ditto

IAM 21 = 50.00% wt. Structure 13a, ditto

IAM 22 = 50.00% wt. Structure 14a, ditto

IAM 23 = 50.00% wt. Structure 14b, ditto

IAM 24 = 50.00% wt. Structure 15a, ditto

IAM 25 = 50.00% wt. Structure 15b, ditto

IAM 26 = 50.00% wt. Structure 33, ditto

IAM 27 = 50.00% wt. Structure 34, ditto

IAM 28 = 50.00% wt. Structure 35, ditto

IAM 29 = 50.00% wt. Structure 36, ditto

IAM 30 = 50.00% wt. Structure 37, ditto

IAM 31 = 50.00% wt. Structure 38, ditto

IAM 32 = 50.00% wt. Structure 39, ditto

IAM 33 = 50.00% wt. Structure 40, ditto

IAM 34 = 50.00% wt. Structure 41, ditto

IAM 35 = 50.00% wt. Structure 42, ditto

IAM 36 = 50.00% wt. Structure 43, ditto

IAM 37 = 50.00% wt. Structure 44, ditto

**IAM 38 = 25.00% wt. Structure 16a + 25.00% wt. Structure 20b + 15.00% wt. coconut oil + 10.00% wt. lecithin + 25.00% wt. water**

IAM 39 = 25.00% wt. Structure 16b + 25.00% wt. Structure 20a, ditto

IAM 40 = 25.00% wt. Structure 22a + 25.00% wt. Structure 28a, ditto

IAM 41 = 25.00% wt. Structure 22b + 25.00% wt. Structure 28b, ditto

IAM 42 = 25.00% wt. Structure 2 + 25.00% wt. Structure 42, ditto

Since acetals dissolve in esters, the addition of coconut oil could be omitted.

**IAM 43 = 40.00% wt. Structure 2 + 10.00% wt. Structure 45 + 10.00% wt. lecithin + 40.00% wt. water**

IAM 44 = 40.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto

IAM 45 = 40.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto

IAM 46 = 40.00% wt. Structure 16a + 10.00% wt. Structure 48, ditto

IAM 47 = 40.00% wt. Structure 17b + 10.00% wt. Structure 50, ditto

**IAM 48 = 30.00% wt. Structure 2 + 10.00% wt. Structure 45 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**

IAM 49 = 30.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto

IAM 50 = 30.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto

IAM 51 = 30.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto

- IAM 52 = 30.00% wt. Structure 5a + 10.00% wt. Structure 49, ditto
- IAM 53 = 30.00% wt. Structure 42 + 10.00% wt. Structure 48, ditto
- IAM 54 = **20.00% wt. Structure 2 + 10.00% wt. Structure 45 + 10.00% wt. Structure 52 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**
- IAM 55 = 20.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto
- IAM 56 = 20.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto
- IAM 57 = 20.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto
- IAM 58 = 20.00% wt. Structure 5a + 10.00% wt. Structure 51, ditto
- IAM 59 = **20.00% wt. Structure 2 + 10.00% wt. Structure 45 + 10.00% wt. Structure 53 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**
- IAM 60 = 20.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto
- IAM 61 = 20.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto
- IAM 62 = 20.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto
- IAM 63 = 20.00% wt. Structure 5a + 10.00% wt. Structure 51, ditto
- IAM 64 = **20.00% wt. Structure 2 + 10.00% wt. Structure 45 + 5.00% wt. Structure 52 + 5.00% wt. Structure 53 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**
- IAM 65 = 20.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto
- IAM 66 = 20.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto
- IAM 67 = 20.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto
- IAM 68 = 20.00% wt. Structure 5a + 10.00% wt. Structure 51, ditto
- IAM 69 = **16.00% wt. Structure 2 + 10.00% wt. Structure 45 + 5.00% wt. Structure 52 + 5.00% wt. Structure 53 + 4.00% wt. Structure 56 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**
- IAM 70 = 20.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto
- IAM 71 = 20.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto
- IAM 72 = 20.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto
- IAM 73 = 20.00% wt. Structure 5a + 10.00% wt. Structure 51, ditto

- IAM 74 = 16.00% wt. Structure 2 + 10.00% wt. Structure 45 + 5.00% wt. Structure 52 + 5.00% wt. Structure 53 + 4.00% wt. Structure 56 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**
- IAM 75 = 20.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto**
- IAM 76 = 20.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto**
- IAM 77 = 20.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto**
- IAM 78 = 20.00% wt. Structure 5a + 10.00% wt. Structure 51, ditto**
- IAM 79 = 16.00% wt. Structure 2 + 10.00% wt. Structure 45 + 5.00% wt. Structure 52 + 5.00% wt. Structure 53 + 2.00% wt. Structure 58 + 2.00% wt. Structure 59 + 10.00% wt. octanoic acid + 10.00% wt. lecithin + 40.00% wt. water**
- IAM 80 = 20.00% wt. Structure 8a + 10.00% wt. Structure 46, ditto**
- IAM 81 = 20.00% wt. Structure 14b + 10.00% wt. Structure 47, ditto**
- IAM 82 = 20.00% wt. Structure 1 + 10.00% wt. Structure 51, ditto**
- IAM 83 = 20.00% wt. Structure 5a + 10.00% wt. Structure 51, ditto**
- IAM 84 = 5.00% wt. Structure 2 + 5.00% wt. Structure 45 + 5.00% wt. Structure 52 + 3.00% wt. Structure 53 + 2.00% wt. Structure 58 + 1.00% wt. Structure 59 + 5.00% wt. octanoic acid + 5.00% wt. coconut oil + 3.00% wt. PEG-40 hydrogenated castor oil + 66.00% wt. water**
- IAM 85 = 5.00% wt. Structure 8b + 5.00% wt. Structure 48 + 5.00% wt. Structure 54 + 3.00% wt. Structure 56 + 2.00% wt. Structure 57 + 1.00% wt. Structure 59 + 5.00% wt. octanoic acid + 5.00% wt. coconut oil + 3.00% wt. PEG-40 hydrogenated castor oil + 66.00% wt. water**
- IAM 86 = 0.20% wt. Structure 1 + 0.80% wt. Structure 45 + 0.67% wt. decyl glucoside + 98.33% wt. water**
- IAM 87 = 0.80% wt. Structure 1 + 0.20% wt. Structure 45, ditto**
- IAM 88 = 0.20% wt. Structure 8a + 0.80% wt. Structure 45, ditto**
- IAM 89 = 0.20% wt. Structure 5a + 0.80% wt. Structure 45, ditto**
- IAM 90 = 0.20% wt. Structure 2 + 0.40% wt. Structure 45 + 0.40% wt. octanoic acid + 0.67% wt. decyl glucoside + 98.33% wt. water**

**IAM 91 = 0.02% wt. Structure 2 + 0.02% wt. Structure 45 + 0.02% wt. octanoic acid  
+ 0.06% wt. decyl glucoside + 99.88% wt. water**

**IAM 92 = 0.04% wt. Structure 1 + 0.02% wt. Structure 46 + 0.02% wt. octanoic acid  
+ 0.06% wt. PEG-40 hydrogenated castor oil + 99.86% wt. water**

**IAM 93 = 0.02% wt. Structure 1 + 0.04% wt. Structure 46, ditto**

**IAM 94 = 0.02% wt. Structure 3 + 0.04% wt. Structure 47, ditto**

### **Examples**

The compositions were tested with regard to their effectiveness as follows.

#### **1. Series of tests for repellence against mosquitoes on people**

The pest repellents according to examples IAM 1 to IAM 85 were each tested on various persons of all age groups, whereby a product on the market, regarded as extremely effective (KIK AKTIV® - with 30% DEET as active ingredient), was used as the reference.

The right lower arm of the respective test person was treated over an area of approx. 250 cm<sup>3</sup> with the appropriate test product (IAM 1 to 85). A quantity of 2 ml of the appropriate test substance was evenly spread over the test area. The treated surface of the lower arm was sealed off, both towards the upper arm and also towards the wrist, with an adhesive tape, sealed against mosquito bites, extending over a short plastic tube. The untreated hand was covered with a thick glove and was thus used at the same time as a control for the biting activity of the mosquitoes, because with the urge to bite they alighted on the glove and tried to bite through the material into the hand located underneath. The left lower arm was similarly treated with the reference product (REF). As experimental animals, about 300 to 400 female yellow fever mosquitoes were put into a breeding cage sized 40x40x40 cm for each single test (IAM on the right against REF on the left). This is a population density which hardly ever occurs naturally and

consequently enables good differentiation of the effectiveness of the individual substances.

For the test the hand and the prepared lower arm were held, after a one-hour waiting period, in the cage for ten minutes every hour, first the left with REF and then the right with IAM and during this period the number of mosquitoes were noted, which

- (a) tried to bite through the glove (positive control),
- (b) flew closer than 3 cm to the treated surface, but turned away again (remote protection effect),
- (c) remained on the treated surface for longer than two seconds, but did not bite and
- (d) bit into the treated skin and sucked blood.

Each test person was subjected to only one experiment each day in order to avoid the risk of product accumulation and any cross-reactions of the products on insufficiently cleansed skin.

Test results:

Legends:

- 1 = Time after the application of the respective test product in hours.
- 2 = Exposure duration within the ten-minute test duration per hour; cancellation on biting and figures for the minutes of the resulting shortened exposure duration.
- a = Mosquitoes resting on the glove.
- b = Flying mosquitoes, which approached the treated surface closer than 3 cm and turned away again, without alighting and mosquitoes which remained alighted on the test surface for less than two seconds.
- c = Mosquitoes which alighted on the treated surface for longer than two seconds, but which did not bite and which thus, together with the mosquitoes of Criterion b, form the annoyance factor.
- d = Number of biting mosquitoes before the cancellation of the test.

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 1				IAM 2				IAM 3				IAM 4			
1	10	130	0	0	0	110	0	0	0	90	0	0	0	90	0	0	0
2	10	130	0	0	0	110	0	0	0	110	0	0	0	120	0	0	0
3	10	130	0	0	0	60	8	2	2(3)*	100	0	0	0	110	0	0	0
4	10	80	5	1	1(1)*					120	0	0	0	120	0	0	0
5	10									120	17	3	0	120	0	0	0
6	10									80	25	10	3(9)*	100	11	8	2(4)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 5				IAM 6				IAM 7				IAM 8			
1	10	130	0	0	0	110	0	0	0	120	0	0	0	100	0	0	0
2	10	110	0	0	0	110	0	0	0	110	0	0	0	120	0	0	0
3	10	110	0	0	0	110	0	0	0	120	0	0	0	120	0	0	0
4	10	110	7	2	0	110	6	2	0	100	6	1	0	110	0	0	0
5	10	100	12	4	0	110	16	5	0	100	13	4	0	110	0	0	0
6	10	70	10	3	1(2)*	80	20	9	2(4)*	80	22	11	3(3)*	50	11	8	2(1)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 9				IAM 10				IAM 11				IAM 12			
1	10	100	0	0	0	100	0	0	0	130	0	0	0	130	0	0	0
2	10	100	0	0	0	100	0	0	0	110	0	0	0	130	0	0	0
3	10	100	0	0	0	110	0	0	0	100	0	0	0	130	0	0	0
4	10	100	0	0	0	120	2	0	0	110	4	2	0	120	15	0	0
5	10	90	9	3	0	100	9	3	0	100	11	5	0	120	15	2	0
6	10	50	17	7	4(1)*	90	18	10	5(1)*	80	21	14	4(2)*	110	31	8	4(2)*



1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 13				IAM 14				IAM 15				IAM 16			
1	10	130	0	0	0	100	0	0	0	120	0	0	0	80	0	0	0
2	10	120	0	0	0	100	0	0	0	90	0	0	0	100	0	0	0
3	10	130	0	0	0	120	0	0	0	90	0	0	0	120	0	0	0
4	10	120	15	1	0	120	22	5	0	90	4	1	0	110	4	0	0
5	10	100	16	2	0	110	22	5	0	100	14	8	0	110	30	5	0
6	10	100	37	17	1(1)*	100	28	22	2(3)*	100	29	10	3(2)*	100	32	18	5(4)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 17				IAM 18				IAM 19				IAM 20			
1	10	80	0	0	0	100	0	0	0	70	0	0	0	130	0	0	0
2	10	100	0	0	0	130	0	0	0	120	0	0	0	100	0	0	0
3	10	110	5	0	0	120	4	0	0	120	0	0	0	120	0	0	0
4	10	120	5	2	0	130	9	6	0	100	10	2	0	130	3	1	0
5	10	120	19	13	3(3)*	110	25	16	4(3)*	110	27	13	0	120	12	6	0
6	10									40	20	11	4(3)*	50	11	8	2(4)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 21				IAM 22				IAM 23				IAM 24			
1	10	80	0	0	0	90	0	0	0	90	0	0	0	130	0	0	0
2	10	80	0	0	0	100	0	0	0	110	0	0	0	130	0	0	0
3	10	100	0	0	0	110	0	0	0	100	0	0	0	100	4	0	0
4	10	100	0	0	0	120	14	6	0	120	0	0	0	100	7	0	0
5	10	100	29	13	0	100	12	6	0	90	37	9	0	120	12	3	0
6	10	100	14	1	1(1)*	80	21	10	3(1)*	40	5	5	3(1)*	20	13	2	2(1)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 25				IAM 26				IAM 27				IAM 28			
1	10	130	0	0	0	130	0	0	0	120	0	0	0	100	0	0	0
2	10	120	0	0	0	120	0	0	0	120	0	0	0	100	0	0	0
3	10	130	4	1	0	130	0	0	0	100	4	0	0	100	3	0	0
4	10	110	15	3	0	100	6	3	0	100	7	0	0	100	8	0	0
5	10	100	12	8	0	100	16	4	0	100	20	7	0	100	17	4	0
6	10	20	7	7	1(1)*	40	5	5	5(1)*	30	2	2	2(1)*	10	15	5	2(1)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 29				IAM 30				IAM 31				IAM 32			
1	10	140	0	0	0	110	0	0	0	120	0	0	0	130	0	0	0
2	10	140	0	0	0	110	0	0	0	120	3	0	0	120	0	0	0
3	10	150	5	0	0	110	3	0	0	120	5	0	0	120	10	0	0
4	10	100	9	1	0	110	13	4	0	120	9	4	0	120	20	5	0
5	10	20	12	5	2(2)*	10	2	2	2(1)*	20	1	1(1)*	0	30	6	3	3(\$)*
6																	

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 33				IAM 34				IAM 35				IAM 36			
1	10	100	0	0	0	100	0	0	0	110	0	0	0	120	0	0	0
2	10	100	0	0	0	90	0	0	0	100	0	0	0	100	0	0	0
3	10	90	0	0	0	90	0	0	0	100	0	0	0	120	0	0	0
4	10	90	0	0	0	100	0	0	0	100	0	0	0	100	10	0	0
5	10	100	4	1	0	100	5	4	0	100	7	3	0	100	30	8	0
6	10	80	6	6	2(4)*	60	8	2	2(3)*	60	10	10	3(1)*	100	15	10	4(2)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 37				IAM 38				IAM 39				IAM 40			
1	10	70	0	0	0	120	0	0	0	90	0	0	0	110	0	0	0
2	10	70	0	0	0	120	0	0	0	90	0	0	0	90	0	0	0
3	10	100	0	0	0	100	0	0	0	90	0	0	0	90	2	0	0
4	10	110	10	1	0	90	11	3	0	100	6	1	0	100	12	3	0
5	10	110	19	8	0	90	15	6	0	100	17	6	0	110	20	6	0
6	10	100	17	7	3(7)*	70	18	5	3(6)*	100	5	5	3(2)*	110	3	3	3(1)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 41				IAM 42				IAM 43				IAM 44			
1	10	100	0	0	0	80	0	0	0	100	0	0	0	120	0	0	0
2	10	110	0	0	0	120	0	0	0	110	0	0	0	120	0	0	0
3	10	120	3	0	0	130	7	0	0	110	0	0	0	120	0	0	0
4	10	110	15	4	0	130	18	4	0	100	0	0	0	120	0	0	0
5	10	110	19	7	0	120	25	10	0	100	1	0	0	90	0	0	0
6	10	60	7	7	1(1)*	50	2	2	2(1)*	110	7	1	0	90	3	1	0
7	10									70	18	6	3(4)*	100	21	8	3(2)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 45				IAM 46				IAM 47				IAM 48			
1	10	130	0	0	0	80	0	0	0	90	0	0	0	100	0	0	0
2	10	120	0	0	0	120	0	0	0	110	0	0	0	90	0	0	0
3	10	120	0	0	0	130	0	0	0	110	0	0	0	100	0	0	0
4	10	120	5	0	0	130	8	4	0	90	0	0	0	90	0	0	0
5	10	100	9	4	0	120	15	10	0	100	1	0	0	90	0	0	0
6	10	110	35	9	0	100	22	12	0	100	7	1	0	90	3	1	0
7	10	30	21	14	4(1)*	50	12	12	2(1)*	70	15	5	3(6)*	90	6	3	0
8	10													50	14	10	5(2)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 49				IAM 50				IAM 51				IAM 52			
1	10	80	0	0	0	100	0	0	0	130	0	0	0	100	0	0	0
2	10	80	0	0	0	120	0	0	0	130	0	0	0	90	0	0	0
3	10	120	0	0	0	110	0	0	0	110	0	0	0	100	0	0	0
4	10	120	0	0	0	110	0	0	0	120	0	0	0	90	0	0	0
5	10	120	0	0	0	110	0	0	0	120	1	0	0	90	0	0	0
6	10	120	5	3	0	110	2	1	0	110	7	1	0	90	3	1	0
7	10	130	11	4	0	110	18	12	0	120	18	6	0	90	6	3	0
8	10	60	8	4	4(2)*	80	10	8	2(1)*	80	12	9	5(2)*	50	15	8	3(3)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 53				IAM 54				IAM 55				IAM 56			
1	10	90	0	0	0	70	0	0	0	80	0	0	0	100	0	0	0
2	10	100	0	0	0	90	0	0	0	100	0	0	0	100	0	0	0
3	10	110	0	0	0	110	0	0	0	100	0	0	0	110	0	0	0
4	10	120	0	0	0	120	0	0	0	100	0	0	0	100	0	0	0
5	10	110	5	0	0	100	0	0	0	90	0	0	0	110	0	0	0
6	10	100	8	2	0	90	0	0	0	110	0	0	0	110	0	0	0
7	10	100	17	8	0	90	5	2	0	100	8	4	0	100	6	1	0
8	10	40	11	10	2(3)*	100	14	3	0	100	15	3	0	100	18	10	
9	10					50	16	8	2(1)*	50	4	4	1(2)*	70	5	5	5(1)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 57				IAM 58				IAM 59				IAM 60			
1	10	60	0	0	0	90	0	0	0	70	0	0	0	50	0	0	0
2	10	60	0	0	0	90	0	0	0	90	0	0	0	90	0	0	0
3	10	100	0	0	0	100	0	0	0	80	0	0	0	100	0	0	0
4	10	130	0	0	0	100	0	0	0	120	0	0	0	130	0	0	0
5	10	100	0	0	0	110	0	0	0	120	0	0	0	120	0	0	0
6	10	110	6	0	0	100	10	0	0	130	0	0	0	120	0	0	0
7	10	110	23	2	0	110	10	1	0	130	24	0	0	120	17	0	0
8	10	100	14	5	0	110	24	5	0	130	30	0	0	100	28	0	0
9	10	90	9	4	2(4)*	80	6	6	3(1)*	100	14	5	1(7)*	80	36	4	2(3)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 61				IAM 62				IAM 63				IAM 64			
1	10	90	0	0	0	100	0	0	0	110	0	0	0	100	0	0	0
2	10	100	0	0	0	90	0	0	0	90	0	0	0	100	0	0	0
3	10	110	0	0	0	110	0	0	0	130	0	0	0	110	0	0	0
4	10	120	0	0	0	120	0	0	0	120	0	0	0	100	0	0	0
5	10	110	10	0	0	100	10	0	0	110	6	0	0	110	0	0	0
6	10	100	12	0	0	90	10	0	0	130	14	0	0	110	0	0	0
7	10	100	20	8	0	100	15	1	0	120	28	0	0	100	6	1	0
8	10	120	35	6	0	100	44	10	0	100	35	3	0	100	18	10	0
9	10	100	10	10	2(1)*	50	50	20	9(1)*	20	10	10	7(1)*	70	5	5	5(1)*

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 65				IAM 66				IAM 67				IAM 68			
1	10	120	0	0	0	100	0	0	0	100	0	0	0	80	0	0	0
2	10	120	0	0	0	110	0	0	0	100	0	0	0	80	0	0	0
3	10	120	0	0	0	120	0	0	0	120	0	0	0	90	0	0	0
4	10	100	0	0	0	130	0	0	0	130	0	0	0	140	0	0	0
5	10	100	12	0	0	140	10	0	0	110	0	0	0	120	6	0	0
6	10	100	28	0	0	110	20	0	0	120	23	0	0	90	12	0	0
7	10	90	37	2	0	110	25	4	0	120	23	0	0	90	17	3	0
8	10	110	41	15	0	120	34	10	0	120	21	7	0	110	15	3	0
9	10	30	11	11	8(1)*	30	12	10	4(1)*	40	14	11	4(1)*	70	24	14	5(4)*

[illegible]

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 73				IAM 74				IAM 75				IAM 76			
1	10	120	0	0	0	80	0	0	0	90	0	0	0	110	0	0	0
2	10	120	0	0	0	80	0	0	0	90	0	0	0	110	0	0	0
3	10	120	0	0	0	100	0	0	0	120	0	0	0	120	0	0	0
4	10	100	0	0	0	100	0	0	0	110	4	0	0	130	0	0	0
5	10	100	0	0	0	90	0	0	0	70	12	2	0	100	20	4	0
6	10	130	22	0	0	100	7	0	0	90	20	3	0	110	23	4	0
7	10	90	35	3	0	90	16	1	0	130	34	6	0	90	17	3	0
8	10	90	33	3	0	100	30	1	0	1010	35	5	0	70	15	7	0
9	10	30	10	10	4(2)*	20	33	1	0	20	44	3	3(1)*	20	4	4	4(1)*
10	10					20	8	7	3(1)*								

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 77				IAM 78				IAM 79				IAM 80			
1	10	100	0	0	0	70	0	0	0	120	0	0	0	60	0	0	0
2	10	90	0	0	0	90	0	0	0	110	0	0	0	90	0	0	0
3	10	100	0	0	0	90	0	0	0	100	0	0	0	100	0	0	0
4	10	100	0	0	0	110	4	0	0	100	0	0	0	130	0	0	0
5	10	100	15	2	0	90	13	2	0	90	6	0	0	100	20	0	0
6	10	110	20	5	0	120	12	4	0	80	13	1	0	110	20	6	0
7	10	100	20	1	0	120	26	3	0	120	21	4	0	120	35	3	0
8	10	100	30	10	0	110	33	9	0	100	25	8	0	100	30	5	0
9	10	30	7	6	2(1)*	50	5	5	5(1)*	30	34	13	3(1)*	50	12	12	6(3)*
10	10									10	10	10	3(1)*				

1	2	a	b	c	d	a	b	c	d	a	b	c	d	a	b	c	d
Product		IAM 81				IAM 82				IAM 83				IAM 84			
1	10	100	0	0	0	100	0	0	0	70	0	0	0	60	0	0	0
2	10	100	0	0	0	80	0	0	0	100	0	0	0	80	0	0	0
3	10	100	0	0	0	100	0	0	0	120	0	0	0	70	0	0	0
4	10	110	0	0	0	90	0	0	0	100	0	0	0	100	0	0	0
5	10	120	0	0	0	90	8	0	0	100	10	0	0	100	0	0	0
6	10	110	28	2	0	110	19	2	0	110	20	2	0	90	4	0	0
7	10	100	27	2	0	100	25	2	0	90	25	6	0	120	25	0	0
8	10	100	30	1	0	110	36	7	0	190	25	5	0	100	25	2	0
9	10	20	30	18	8(1)*	20	9	9	3(1)*	40	15	13	6(1)*	100	25	2	0
10	10													40	22	5	3(3)*

1	2	a	b	c	d	a	b	c	d
Product		IAM 85				IAM 86			
1	10	70	0	0	0	70	0	0	0
2	10	70	0	0	0	80	0	0	0
3	10	80	0	0	0	90	0	0	0
4	10	100	5	0	0	130	0	0	0
5	10	120	10	1	0	130	0	0	0
6	10	120	20	0	0	120	15	2	0
7	10	110	25	0	0	110	15	6	0
8	10	80	37	5	0	100	30	4	0
9	10	20	9	9	4(2)*	40	9	8	2(1)*



## **2. Series of tests for repellence against ticks on guinea pigs**

On a total of twelve adult, female guinea pigs the right side was shaved to an extent of about 4x4 cm and the skin and surrounding hairy locations were treated manually with in each case approx. 2 ml of the respective test product, covering the full surface. Then each hour, one adult tick in each case of the genus common tick (*Ixodes ricinus*), raised in the laboratory and kept hungry for two weeks, was placed in the centre of the treated, shaved area using tweezers and its behaviour was observed. The guinea pigs were not sedated as is usual, but instead, to simulate as natural a situation as possible, were fed with lettuce and they remained rather calm, because they are used to a human environment.

It was immediately noticeable that the ticks did not, as on the untreated guinea pig serving as the control animal (CA), start to wander around looking for a suitable place to bite into (normally a bending fold under the rear legs), but rather moved over the treated surface in a tight circle for a few minutes and then, with the slightest movement from their hosts, fell to the bottom of the cage, although ticks normally can find a hold with their holding claws on even smooth surfaces. The fallen ticks were immediately placed in labelled jars and observed over 72 hours. After this time period all observed ticks were dead.

### **List of test individuals and test products:**

- the untreated controls were labelled with CA;
- the experimental animal treated with the reference product ANTI BRUMM with REF; and
- the experimental animals treated with the compositions according to the invention were labelled with IAM 1, IAM 4, IAM 38, IAM 43, IAM 48, IAM 54, IAM 59, IAM 64, IAM 74 and IAM 84.

**Results of effectiveness:**

**CA:** after a short orientation phase, each of the ticks placed in location hourly and individually within the test time over 8 hours, soon began to bore and suck blood, whereupon they were immediately removed so as not to weaken the control animal.

**REF:** the ticks during the first 2 hours fell down as with the products of the IAM series and were dead within 72 hours. The tick applied in the 3<sup>rd</sup> hour began to bore down, whereupon the test was interrupted and the protection time was therefore 2 hours.

**Protection times for the compositions according to the invention against ticks on guinea pigs:**

**X = Protection time**

**Y = Time of the first boring down of a freshly applied tick**

Product	IAM 1	IAM 4	IAM 38	IAM 43	IAM 48	IAM 54	IAM 59	IAM 64	IAM 74	IAM 84
1 hr.										
2 hrs.										
3 hrs.	x									
4 hrs.	y		x		x					
5 hrs.			y		y	x	x		x	
6 hrs.				x		y	y	x	y	
7 hrs.		x		y				y		x
8 hrs.		y								y

All ticks which fell off without attempting to bore down were dead within the observation period of 72 hours.

**3. Series of tests for repellence against ticks on the dog**

To check the repellent effect against ticks as a typical member of the Acarina and which also represent the most dangerous and widespread member of this genus for people and animals, a five-day free ranging test was carried out. These experiments were not carried out on people for ethical reasons.

Six dogs from an animal home and of mixed breed and sex, but of approximately the same weight were made available together with two dog handlers from the group of personnel, who each over the five days took three of the dogs for a walk each day for 4 hours in a known tick infested region (Föhrenwald on the outskirts of the Vienna new town). Daily in always the same way, the two dogs labelled V1 and V2 were treated with IAM 64, with 10 ml being worked into the dog's coat in each case using a brush. The two dogs labelled V3 and V4 were treated as described above with IAM 79, the dog REF with Kik aktiv and the dog K (control) always remained untreated. The two dog handlers were treated daily on the complete naked body with 10 ml of IAM 84 before they went out, because although they were inoculated against spring-summer encephalitis, the risk of transmission of Lyme disease by tick bites was to be avoided and at the same time the protection time of eight hours against tick bites previously found in the laboratory on shaven guinea pig skin was to be confirmed over 4 hours on humans who were potentially at risk.

As an additional measure for reducing the risk, shoes, socks, long trousers and the dog handlers' jackets, which were closed around the arms and legs by pull cords were sprayed daily with in each case 5 ml of IAM 84 giving full coverage. After each of the daily four-hour walks, the animals and their handlers were examined meticulously for ticks which were documented and collected in labelled jars and observed for any fatalities over 72 hours.

### **Results of effectiveness:**

#### **a) Handler 1, Dog K and Dogs V1 and V2**

Over the five days no ticks were found on the skin of Handler 1 and from the clothing a total of four 4 ticks were removed, which in contrast to Dog K had not worked their way through to the skin, but which already proved to be immobile on removal from the clothing and which did not recuperate over the observation period of 72 hours, whereby the fatal effect of IAM 84 on ticks already noticed in the laboratory test was confirmed.

A total of 66 ticks were removed from the untreated control dog (K) over the five days with all ticks surviving the following observation period, whereby it was established that ticks were present and able to live in the region.

No ticks were found on V1 and V2.

b) Handler 2, Dog REF and Dogs V3 and V4

No ticks were found on the clothing or skin of Handler 2 and also V3 and V4 were free of ticks, whereas on the coat of REF over the five days a total of 23 ticks were found which survived the following observation time of 72 hours also without any problem, because they were then removed from the jars and placed on the shaved side of a guinea pig in the laboratory where they immediately looked for a suitable location to bite and immediately on reaching the hair covering they started to bore down, whereupon they were removed with tweezers dipped for five minutes in REF according to the rules of the immersion test of the ectoparasite screening and after 72 hours of a further observation period were again applied to the guinea pig where they again started to bore down.

The result showed that although REF had an acceptable repellence to ticks, it exhibited no fatal effect. The 66 ticks removed from the untreated Dog K were dipped for five minutes in IAM 79 and were dead within thirty minutes (they also did not revive 72 hours later).

#### **4. Use with insects**

##### **A. Reference products used (REF)**

- |      |                                |                              |
|------|--------------------------------|------------------------------|
| (I)  | 0.20% wt. methomyl (carbamate) |                              |
|      | 99.80% wt. water               |                              |
| (II) | 0.50% wt. chlorpyrifos         | (III)                        |
|      | 0.37% wt. neopynamin           | 20.00% wt. deltamethrin      |
|      | 1.95% wt. methoxychlor         | 10.00% wt. water             |
|      | 97.18% wt. water               | 0.20% wt. carbopol           |
|      |                                | 69.80% wt. 1,2,3-propantriol |

**B. IAM 86 and IAM 90** were used comparatively in equal quantities to REF (I) and (II) in the laboratory experiment as spray agents for use against the following pests (adults and metamorphoses):

- A) *Musca domestica* - Common House Fly
- B) *Blatella germanica* - German Cockroach
- C) *Lasius niger* - Black Garden Ant
- D) *Ixodes ricinus* - Common Tick
- E) *Tineola bisselliella* - Common Clothes Moth

For the tests 30 samples each of the flying pests A) and E) were put into a cage covered with gauze with a size of 1 x 1 x 1 m and each was sprayed with 1 ml of IAM 86 and IAM 90 and REF (I) and REF (II). The animals incapable of flight and lying on the bottom were evaluated after respectively 1, 5, 10 and 20 minutes, as was the mortality or revival rate (recuperation) after 24 hours.

Flies						Moths				
	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
IAM 86	100	100	100	100	100	100	100	100	100	100
IAM 90	100	100	100	100	100	100	100	100	100	100
REF (I)	20	80	100	100	95	100	100	100	100	100
REF (II)	10	70	100	100	100	100	100	100	100	100

Of the creeping pests B), C) and D) 30 samples in each case of mixed metamorphoses were placed in a bath of size 1 x 1 x 1 m and sprayed with 1 ml of IAM 86 and REF (I).

IAM 86						REF (I)				
Cock-roaches	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	100	100	100	100	100	90	90	100	100	100
Ants	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	100	100	100	100	100	90	90	90	100	100
Ticks	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	40	60	100	100	100	0	0	20	40	100

The evaluation after the respective expired time acquired exclusively animals lying on their backs which showed no leg or antenna movement under the magnifying glass.

Also these experimental setups showed that with the use of the insect repellent according to the invention surprisingly not only a comparable, unexpected effect could be achieved, but rather an effect could be achieved which was even better than with the normal marketed comparative product (REF I).

### **Long-term test**

The effectiveness of a permanent coating against cockroaches was assessed in a long-term test over six months. A flat stainless-steel bath with dimensions L=50 cm, W=30 cm, H=2 cm was coated in each case with 2 ml of IAM 1 and 2 ml of REF (III) around the 2 cm high side surfaces and on the bottom and placed in each case in large acrylic glass baths (1 x 1 x 1 m). Then the following were placed in the treated stainless steel baths:

Once per month for a total of six consecutive months in each case ten mixed examples (adults and nymphs) of

*Blatella germanica* - German Cockroach

*Periplaneta americana* - American Cockroach and

*Blatta orientalis* - Oriental Cockroach

whereby these were then transferred after product contact into the untreated acrylic baths. Then the treated bath was removed and kept for the other experiments. The contaminated cockroaches were supplied with water and food in the untreated bath and observed. After respectively 5, 10, 30 minutes and 24 hours, the animals lying on their backs were counted and after a further 24 hours observation period the mortality rate as a percentage of the total number of cockroaches used was determined.

**Blatella germanica**

IAM 1						REF III				
	Minutes			Hours			Minutes			
	5	10	30	24	48		5	10	30	
Month 1	100	100	100	100	100	100	100	100	100	100
Month 2	100	100	100	100	100	100	100	100	100	100
Month 3	100	100	100	100	100	100	100	100	100	100
Month 4	100	100	100	100	100	100	100	100	100	100
Month 5	90	90	90	100	100	90	90	90	90	100
Month 6	60	70	85	100	100	60	70	70	100	100

**Periplaneta americana**

IAM 1						REF III				
	Minutes			Hours			Minutes			
	5	10	30	24	48		5	10	30	
Month 1	100	100	100	100	100	100	100	100	100	100
Month 2	100	100	100	100	100	100	100	100	100	100
Month 3	100	100	100	100	100	100	100	100	100	100
Month 4	100	100	100	100	100	100	100	100	100	100
Month 5	80	90	100	100	100	80	100	100	100	100
Month 6	50	60	90	100	100	60	60	60	100	100



**Blatta orientalis**

IAM 1						REF III				
	Minutes			Hours		Minutes			Hours	
	5	10	30	24	48	5	10	30	24	48
Month 1	100	100	100	100	100	100	100	100	100	100
Month 2	100	100	100	100	100	90	100	100	100	100
Month 3	100	100	100	100	100	85	100	100	100	100
Month 4	100	100	100	100	100	60	80	100	100	100
Month 5	90	90	90	100	100	0	35	60	90	100
Month 6	60	70	85	100	100	0	30	60	85	100

This long-term experiment shows the surprisingly good and long-lasting effect of the insect repellent used according to the invention, in particular in its 100% effectiveness with mixed species and stages of cockroaches (Blattodea), also after a storage time of six months.

### **5. Use on vegetable plants and flowers in nurseries**

#### **Test AI:**

IAM 91 and IAM 92 were compared with REF (I) and REF (II). To do this, in each case 20 begonias, severely infested with red mites (*Panonychus ulmi*), were completely wetted with a quantity of 20 ml in each case (1 ml per plant) of the respective products.

#### **Results:**

With IAM 91 and with IAM 92 as well as with the application of REF (II) all plants were completely free of any infestation at the bonitures after 1, 4, 7 and 14 days. With REF(I) the bonitures were free of infestation only after one day. After 4 days 10% of the initial infestation was counted, after 7 days 50% and after 14 days 100%.

The evaluation of the phytotoxicity of the products after the application (leaf-edge discoloration, necrosis, etc.) after 14 days showed that with IAM 91 and IAM 92 the value 0 was found on the 5-division scale (no visible changes), with REF (I) and REF (II) the value 1 (slight discoloration of the leaves and blooms).

Thus it was shown that the combinations according to the invention had the same effect as the market products REF (I) and REF (II), however without their risks to the health of the users and plants.

### **TEST AII:**

The same products as in the Test AI were tested in four greenhouses on in each case 200 vegetable plants which were strongly infested with white fly (*Aleyrodes proletella*) and various types of aphids (*Aphididae*).

The applied quantity in each case was 200 ml, i.e. 1 ml per plant, fully wetted.

### **Results:**

With IAM 91 and with IAM 92 as well as with the application of REF (I) and REF (II) all plants were completely free of any infestation in the bonitures after 1, 4, 7 and 14 days.

### **6. Further applications on insects:**

IAM 87 and IAM 88 were used comparatively in equal quantities to REF (I) and REF (II) in the laboratory experiment as spray agents for use against the following pests (adults and metamorphoses):

- A) *Musca domestica* - Common House Fly
- B) *Blatella germanica* - German Cockroach
- C) *Lasius niger* - Black Garden Ant
- D) *Ixodes ricinus* - Common Tick
- E) *Tineola bisselliella* - Common Clothes Moth

For the tests 30 samples each of the flying pests A) and E) were put into a cage covered with gauze with a size of 1 x 1 x 1 m and each was sprayed with 1 ml of the above quoted products. The animals incapable of flight and lying on the bottom were evaluated after respectively 1, 5, 10 and 20 minutes and also the mortality or revival rate (recuperation) after 24 hours.

#### Results in percentage mortality:

Flies						Moths				
	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
IAM 87	100	100	100	100	100	100	100	100	100	100
IAM 88	100	100	100	100	100	100	100	100	100	100
REF (I)	20	80	100	100	95	100	100	100	100	100
REF (II)	10	70	100	100	100	90	100	100	100	100

For the creeping pests, similarly in each case 30 samples of mixed metamorphoses were placed in baths with dimensions 1 x 1 x 1 m and in each case sprayed with 1 ml - boniture as before.

IAM 87						REF(I)				
Cock-roaches	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	100	100	100	100	100	60	90	100	100	100
Ants	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	85	100	100	100	100	10	35	70	100	100
Ticks	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	40	60	100	100	100	0	0	20	40	100

IAM 88						REF (II)				
Cock-roaches	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	100	100	100	100	100	65	95	100	100	100
Ants	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	85	100	100	100	100	10	30	85	100	100
Ticks	1	5	10	20 min.	24 hrs.	1	5	10	20 min.	24 hrs.
	55	100	100	100	100	0	25	60	100	100

The evaluation after the respective expired time acquired exclusively animals lying on their backs which showed no leg or antenna movement under the magnifying glass. Also these experimental setups showed that with the use of the insect repellent according to the invention surprisingly not only a comparable, expected effect could be achieved, but rather an effect could be achieved which was better than with the normal marketed comparative products REF (I) and REF (II).